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IS 7910 (2003): Monoethanolamine [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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भारतीय मानक
मोनोइथनोलअमीन — विशिष्टि
(पहला पुनरीक्षण)

Indian Standard
MONOETHANOLAMINE — SPECIFICATION
(*First Revision*)

ICS 71.080.20

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Including Aromatic Hydrocarbons Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1975. The Committee responsible for the preparation of this standard felt that the standard should be revised with a view to up date it in accordance with the advancement in technology. In this revision the requirements of moisture content, distillation range have been modified. An additional, instrumental GLC method has been incorporated for determination of purity, as a result of which the impurities can also be determined accurately.

Monoethanolamine finds extensive use in the acid gas removal processes of the fertilizer industry, refinery and dry ice manufacturing industry. In the pharmaceutical industry, it is used in the manufacture of penicillin, piperazine and anesthetics. It is being used in the manufacture of detergent powders, surface coatings and paints. It also finds wide applications in textiles, dyestuff industry, in the scouring oils, viscose yarn spinning and anti-rust compound used in the formulation of lubricating oils.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

MONOETHANOLAMINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for monoethanolamine.

2 REFERENCES

The following standards contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent revisions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
2362 : 1993	Determination of water by the Karl Fischer method (<i>first revision</i>)
4730 : 1994	Methods for determination of density of liquids (<i>first revision</i>)
4905 : 1968	Method for random sampling
5298 : 1983	Method for determination of distillation range and of distillation yield (<i>first revision</i>)
5717 : 1991	Pyknometers (<i>first revision</i>)
5813 : 1970	Method for determination of crystallizing point
8768 : 2000	Method of measurement of colour in liquid chemical products platinum-cobalt scale (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

The material shall mainly consist of monoethanolamine ($\text{H}_2\text{NCH}_2\text{CH}_2\text{OH}$) and may contain small amounts of diethanolamine and triethanolamine. It shall be a clear liquid with a slight characteristic odour.

3.2 Miscibility

It shall be miscible with water and with ethanol (96 percent) in all volumes and slightly soluble in ether.

3.3 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Monoethanolamine
(Clause 3.3)

SI No.	Characteristic	Requirement	Methods of Test, Ref to	
			Annex (4)	IS No. (5)
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 20/20°C	1.015 to 1.020	—	4730
ii)	Moisture content, percent by mass, <i>Max</i>	0.5	A	2362
iii)	Equivalent mass	61 to 62.5	B	—
iv)	Distillation range :		—	5298
	a) Initial boiling point at 760 mm Hg, <i>Min</i> , °C	168	—	—
	b) Dry point at 760 mm Hg, <i>Max</i> , °C	178	—	—
v)	Crystallizing point, <i>Min</i> , °C	9	—	5813
vi)	Colour, Hazen units, <i>Max</i>	25	—	8768
vii)	Assay, percent by mass, <i>Min</i>	98.0	C	—
viii)	Impurities, percent by mass, <i>Max</i>	2.0	C (Method 2)	—

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in airtight mild steel drums.

4.2 Marking

Each container shall be securely closed and shall bear legibly the following information:

- Name of the material;
- Net mass of the material;
- Name of the manufacturer and his recognized trade-mark, if any; and
- Lot or batch or drum number in code or otherwise.

4.2.1 BIS Certification Marking

The container may also be marked with the Standard Mark.

4.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standard Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

Representative samples of the material shall be drawn and their conformity to the requirements of this standard judged as prescribed in Annex D.

6 TEST METHODS

The tests for the requirements listed under 3 shall be carried out according to the methods prescribed in Annex A to C and referred Indian Standards under col 5 of Table 1.

7 QUALITY OF REAGENTS

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the result of analysis.

ANNEX A

[Table 1, *Sl No.* (ii)]

DETERMINATION OF MOISTURE CONTENT

A-1 OUTLINE OF THE METHOD

Moisture is determined by the Karl Fischer method using electrometric end-point.

A-2 PROCEDURE

Carry out the determination as prescribed in IS 2362.

Transfer 75 ml of methanol to the titration flask. Add 15 ml of glacial acetic acid and titrate with the Fischer reagent to the end-point. To the titrated liquid quickly add an accurately weighed quantity of the material, stir vigorously and again titrate with the reagent. Determine the moisture content of the resulting solution by the procedure given in IS 2362.

ANNEX B

[Table 1, Sl No. (iii)]

DETERMINATION OF EQUIVALENT MASS

B-1 REAGENTS

B-1.1 Standard Hydrochloric Acid — 0.5 N
(see IS 265).

B-1.2 Methyl Red Indicator Solution — 0.1 percent.

B-2 PROCEDURE

Weigh accurately 2 g of the sample and transfer into a 250-ml conical flask. Add 100 ml of water and mix until the solution is homogeneous. Titrate the contents with standard hydrochloric acid using methyl red as an indicator. Similarly, carry out a blank titration.

B-3 CALCULATION

$$\text{Equivalent mass} = \frac{(100 - H) \times M \times 10}{(V_1 - V_2) \times N}$$

where

H = percentage moisture in the sample (see A-2),

M = mass, in g, of the sample taken for the test,

V_1 = volume, in ml, of standard hydrochloric acid used in the titration with the material,

V_2 = volume, in ml, of standard hydrochloric acid used in the blank, and

N = normality of standard hydrochloric acid.

ANNEX C

[Table 1, Sl No. (vii and viii)]

DETERMINATION OF MONOETHANOLAMINE CONTENT

C-1 GENERAL

Two methods have been prescribed for the determination of monoethanolamine.

C-2 METHOD 1 — HEMPEL PIPETTE VOLUMETRIC METHOD

C-2.1 Reagents

C-2.1.1 Sodium Nitrite Solution — 20 percent.

C-2.1.2 Potassium Permanganate Solution —
Dissolve 25 g of potassium hydroxide and 50 g of potassium permanganate in one litre of distilled water.

C-2.1.3 Glacial Acetic Acid

C-2.2 Procedure

Assemble the apparatus as shown in Fig. 1. Add enough water containing 1 percent sulphuric acid solution to the gas burette F and alkaline permanganate solution to the Hempel pipette. Turn stopcock 1 to connect the gas burette with the Hempel

pipette. With the help of the levelling bulb, draw the permanganate solution into the Hempel capillary until it reaches an arbitrary reference point close to the junction of capillary with the gas burette. Turn stopcock 1 through 180°C to connect the gas burette with stopcock 2 which is open to the air.

Adjust the water level in the gas burette F with the help of levelling bulb till the water just comes out of the capillary L below stopcock 2 and close stopcock 1 and 2, so that 2 is open to both air and reaction vessel D . Close stopcocks 3 and 5 and introduce into the funnel A , 10 ml of acetic acid. Open stopcock 5 and let the acetic acid run into the reaction vessel D . Similarly introduce 20 ml of sodium nitrite solution permitting it to run into D . Close stopcock and shake the reactor assembly for 5 min to expel the air from D through L with the help of the evolved oxides of nitrogen. Turn stopcock 1 and 2 so that D and F are connected and add an appropriate amount of sample (10 ml = 0.05g) from B . Close stopcock 3 and shake the reactor for 20 to 25 min.

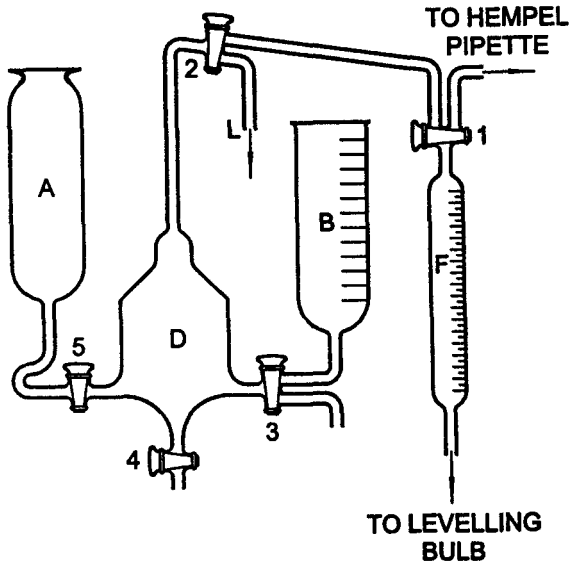


FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF MONOETHANOLAMINE

Collect the gas in gas burette by lowering the levelling bulb and, if necessary absorb the gas in the Hempel pipette so that pressure is not developed in the gas burette. Add water in the bulb A, open stopcock 5 and lower the levelling bulb so that the water level in D rises and comes to the capillary near to the gas burette and all the gases in D are brought to the burette. Connect the gas burette with the Hempel pipette and force the gas into the pipette by means of levelling bulb. Allow stopcock 5 to remain open during this and subsequent operations in order to permit displacement of liquid in D by the oxides of nitrogen formed in the interval. Shake the Hempel pipette to absorb the oxides of nitrogen. Return the remaining gas to the burette bringing the permanganate solution to the previous reference mark in capillary. Balance and read the gas volume in the burette and again return the gas to the Hempel pipette.

Repeat the procedure shaking the residual gas continuously with permanganate until no further absorption is noted and a constant reading is obtained in the burette. Read and record the final residual volume in the burette, the ambient temperature and atmospheric pressure. Run a blank in the same way without the sample.

C-2.3 Calculation

Monoethanolamine content, percent by mass =
$$\frac{(V_1 - V_2) (P_1 - P_2) 0.09797}{(273 + t) M}$$

where

- V_1 = volume, in ml, of gas evolved with the sample;
- V_2 = volume, in ml, of gas evolved during the blank run;
- P_1 = atmospheric pressure, in mm, Hg;
- P_2 = aqueous vapour pressure at t °C, in mm, Hg;
- t = ambient temperature in °C; and
- M = mass, in g, of the sample taken for the test.

C-3 METHOD 2 — GC METHOD

C-3.1 Outline of the Method

About 3 g of the sample is dissolved in 100 ml of distilled water and 0.2 ml of the sample is introduced into the separating column of a gas chromatograph by means of a syringe. The vapourized sample is swept through a packed column by a flow of carrier gas and as each component emerges, it is detected by flame ionization detector and recorded as a peak in a chromatogram. Under the operating conditions the impurities are identified by their relative retention and are determined from their peak areas taking their respective response factors into consideration. The chromatographic conditions and the chromatogram (Fig. 2) given below are for guidance only.

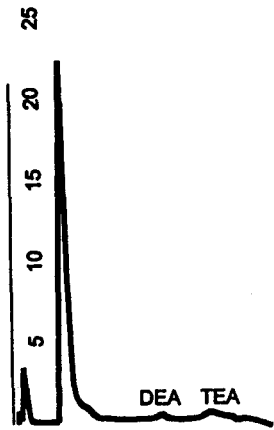


FIG. 2 TYPICAL CHROMATOGRAM FOR MONOETHANOLAMINE

C-3.2 Column — Packed

Material	: Glass
Length	: 107 cm
O D	: 3.2 mm
Packing material	: TENAX-GC of 60/80 mesh.

C-3.3 Reagents**C-3.3.1 Monoethanolamine** — Purity 99 percent.**C-3.3.2 Pure diethanolamine** — 99 percent.**C-3.3.3 Pure triethanolamine** — 99 percent.**C-3.4 Operating Conditions**

Detector	: FID
Injection port temperature	: 280°C
FID temperature	: 280°C
<i>Temperature programming</i>	
Monoethanolamine	: 120 – 190°C
Temperature increasing rate	: 8°C/min
Carrier gas	: Nitrogen
Flow rate	: 20 ml/min
Chart speed	: 1 cm/min
Sample size	: 0.2 µl

C-3.5 Fuel Gas

Hydrogen and zero air flow rates for the flame ionization detector shall be adjusted to the levels recommended by the instrument manufacturer.

C-3.6 Calculation

The total area of secondary peaks should be less than 2 percent of the main peak area, while calculating the area-response factors.

C-3.7 Determination of Response Factor

Prepare a mixture of pure monoethanolamine, pure diethanolamine and pure triethanolamine of known concentration using pure chemicals and inject a suitable sample to take a chromatogram using the operating conditions specified above and determine the area of each peak and then knowing the amount of each component the response factors are determined.

ANNEX D*(Clause 5)***SAMPLING****D-1 GENERAL REQUIREMENTS FOR SAMPLING**

D-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

D-1.2 The sampling instrument shall be clean and dry.

D-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

D-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

D-1.5 The samples shall be placed in suitable, clean, dry and airtight glass bottles or other suitable containers on which the material has no action.

D-1.6 The sample containers shall be of such a size that they are almost three-fourths filled by the sample.

D-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given in 4.2.

D-2 SCALE OF SAMPLING**D-2.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

D-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

D-2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

D-2.3.1 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used (*see* IS 4905). In case random number tables are not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3,....., up to r and so on, r being the integral part of N/n (N being the lot size and n the number of containers to be selected). Every r^{th} container thus counted shall be withdrawn to constitute the sample.

Table 2 Number of Containers to be Selected from Lots of Different Sizes
(Clause D-2.3)

Sl No.	Lot Size N	Sample Size n
(1)	(2)	(3)
i)	3 to 15	3
ii)	16 to 40	4
iii)	41 to 110	5
iv)	111 to 180	6
v)	181 to 300	7
vi)	301 to 500	8
vii)	501 to 800	9
viii)	801 and above	10

D-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

D-3.1 From each of the containers selected according to **D-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. These samples shall constitute individual samples.

D-3.2 From each of these individual portions (see **D-3.1**), an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be divided into three parts, one for the purchaser, one for the supplier and the third for the referee.

D-3.3 Referee Sample

The referee sample consists of the composite sample marked for this purpose, and shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the two and shall be used in case of dispute.

D-4 NUMBER OF TESTS

Tests for the determination of all the characteristics given in Table 1 shall be carried out on the composite sample.

D-5 CRITERIA OF CONFORMITY

For declaring the conformity of the lot the requirements of each of the characteristics, the test results on the composite samples shall meet the corresponding requirements specified.

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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Amendments Issued Since Publication

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